EP102

ELEMENTS OF MATERIAL SCIENCE

LABORATORY MANUAL

Lecturer/Instructor :________________________________

Name: ____

Student ID: ____

Course: ____

FACULTY OF ENGINEERING, ARTCHITECTURE &
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UCSI UNIVERSITY
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1 GENERAL INSTRUCTIONS

The experiments in this manual are designed to introduce to engineering students aspects of material science including atomic structure, material properties and testing. These experiments are also intended to teach students the principles of laboratory protocol and reporting.

Students are required to carry out all the experiments following the general methods and procedures presented in this manual. All experiments are to be completed within the allotted time. Students are expected to be prepared prior to coming for the laboratory session. This includes reading the lab procedure thoroughly and understanding the objectives, operations and hazards that may be encountered during the experiment.

The laboratory experiments shall be conducted in groups. Thus, students have to learn to organize themselves within the groups and to allocate responsibilities among themselves.

2 LABORATORY PROCEDURE AND CONTROL

2.1 Logbook

You are required to keep a logbook for experiments. This must be a bound logbook. You are required to translate the procedure of each experiment in the laboratory manual into graphical illustrations or a flowchart in your logbook before you start.

Record in your logbook every action and observation as you carry out the experiment. Records should be as brief, but concise, as possible.

Each experiment in the logbook should contain the following information:

(i) Title of the experiment
(ii) Date of the experiment carried out
(iii) Objective of the experiment
(iv) Hypothesis of the experiment
(v) Procedure outline or brief description of the method
(vi) Observation (should be recorded throughout the experiment)

A logbook should be neat. Clarity of procedure and results are important. Always use a pen to write on your logbook. Mistakes made should be crossed out clearly. Use of liquid paper or eraser is not allowed.

The lab instructor/lecturer will inspect and sign your logbook during the lab session and possibly grade them at the end of the semester.

2.2 Laboratory group

You will be assigned into a group (please refer to the student notice board or outside the lab after the first session of the laboratory meet up). Students are not allowed to change groups. You are required to work with your group members as much as possible in terms of understanding the experiment and sharing information on the different activities performed by different members. At the end of each of the session, each of the group members is required to carry out peer evaluation. Peer evaluation is private and confidential. The purpose of peer evaluation is to ensure that all group members contribute throughout the laboratory session.

2.3 Laboratory Reports

Each student will be responsible for preparing an individual report after completing each laboratory experiment.

(a) Report submission

Reports must be submitted **one (1) week** after the scheduled experiment and within the first ten (10) minutes of the due lab period. Any unforeseen circumstances must be reported to the lab instructor immediately. Late submissions will not be accepted.

(b) Report style/format

Reports may be hand-written or typed-written. If typed-written, text shall be double spaced, using font type New Times Roman and font size 12. It is recommended that an equation editor be used, otherwise, equations may be handwritten. Atomic structures and other figures may be drawn by hand.

The report shall be:

(i) Written in the passive tense. Example: “Five milligrams of solid was weighed.”

(ii) Graphs must be complete with title, axis labels with units and legend where necessary. The scale of the graph should be appropriate for the data obtained.

(iii) Plagiarism will not be allowed or tolerated.

(c) Report contents

Each report should contain the following:

(i) *Front page* (Refer to Appendix A)

(ii) *Table of contents* (Refer to Appendix B)

(iii) *Introduction/background of the experiment*

Contains a summary of the experiment that is carried out. This section includes the statement of purpose or objectives of the experiment and an introduction to the technique used. It is not necessary to include a discussion of the basic theory relevant to the experiment. The introduction of the report should not be more than 20% of the overall report.

(iv) *Material and Methodology*

This is a brief description of the experimental approach used. It should include a brief description of the test apparatus and instrumentation used, as well as the procedure used to obtain the test results. Summarize the detailed steps used; do not repeat the steps given in your lab experiment.

(v) *Results and discussion*

Test results obtained from the experiment shall be presented in the applicable format: Narrative, tabular, graphical, photographic, or a combination of the above. Numerical data must be presented realistically with appropriate number of decimal places. Data should be tabulated neatly showing raw experimental data, if necessary, and reduced (useful) data. Calculations for reduced data should be briefly described and need not be worked out. Identify formulas, charts, tables or handbooks that were used to obtain the final result. If needed, you may refer sample calculations to the appendix. Any questions given in the lab sheet should be answered in the Discussion section.

(vi) *Conclusion*

This section summarizes your findings of the experiment. If results differ from those expected, provide an explanation based on an analysis of the data. Examine any assumptions used that could be adversely affecting the results.

(vii) *Limitation of the Experiment/Difficulties Encountered (if any)*

Consider any limitations or difficulties encountered during the experiment. Include also any errors that may have occurred. Suggest ways for improvement.

(viii) *Reference*

A minimum of three (3) references should be cited in your report. Refer to Appendix C for the format of citations.

(ix) Appendix (if any)

2.4 Assessment

The laboratory component accounts for 10% of the total marks for the subject. Marks for each report shall be allocated as follows:

Introduction/Background	3%
Material & Methodology	2%
Results & Discussion	5%
Conclusion	3%
Limitation of the experiment	2%
Reference	2%
Format	2%
Peer rating	1%

3 Laboratory Safety

All safety requirements as specified in this section will be strictly enforced. Students are required to comply with the following rules when in the lab.

1. Wear closed shoes and lab coats at all times while in the laboratory. Eye protection (goggles) should be used when necessary.
2. Wear suitable attire for lab work. Do not wear loose or bulky clothes, ties or jewelry when working around rotating equipment. Students with long hair must tie their hair before entering the lab. Open-toe shoes and sandals are not permitted. *(Lecturer/instructor reserves the right not to allow students who do not follow this rule to enter the lab).*
3. Eating and drinking in the lab are strictly prohibited.
4. All hand phones must be switched off.

5. Always be punctual. Students later than 15 minutes are not allowed to perform the experiment.
6. Students must have a towel (for cleaning purposes), a logbook and their lab manual during the lab session.
7. Always follow the instruction of the lab instructor or lecturer before the start of the lab.
8. Clean the bench and return all apparatus to their respective places before you leave the lab.
9. Use only required amounts of reagents to avoid wastage and excess.
10. Dispose of excess reagents as instructed, never return excess reagents into the bottle. Some used chemicals may be washed down the drain, while others require a different method of disposal. If a chemical can go in the sink, be sure to wash it away thoroughly rather than risk an unexpected reaction between chemical ‘leftovers’ later.
11. Make sure you know the location of the following safety devices: eyewash station, emergency shower, fire extinguisher, fire alarm station, emergency evacuation route.
12. Handle all equipment with care to avoid damage during experimental operations.
13. Clean up broken glass and report to the lab instructor or lecturer immediately.

EXPERIMENT 1: IDENTIFICATION OF ENGINEERING MATERIALS

Objective

The objective of this experiment is to enable students to familiarize themselves with different types of engineering materials, compare their physical properties and relate these properties to practical applications.

Introduction

Materials are substances of which something is composed or made. All engineering disciplines involve the use of materials from manufacturing to processing as well as design of components. Some of the commonly encountered materials are wood (timber), concrete, brick, steel, plastic, glass, rubber, aluminium, copper, paper and ceramic. Because of constant research and development, new materials are frequently being created.

Generally, materials are classified into the following groups: metals, polymers and ceramics. Materials in each of these groups possess different structures and properties. In selecting materials for applications, properties that are considered include physical, mechanical, electrical, thermal, magnetic, optical and corrosion (chemical).

Metals are composed of one or more metallic elements and have a crystalline structure. Metals in general are good thermal and electric conductors. They are relatively strong and ductile at room temperature. Metals may be further classified as ferrous or non-ferrous. Most polymeric materials consist of carbon-containing long molecular chains or networks. Their strength and ductility varies greatly. Most polymeric materials are good insulators, have low densities and relatively low softening temperatures. Ceramic materials consist of metallic and non-metallic elements chemically bonded together. Ceramics are strong and hard but also very brittle. They also have low electrical and thermal properties. Semi-conductors are not a major type of material but are considered due to engineering importance. Examples are silicon and germanium. Their electrical characteristics can be modified and controlled; hence, they are widely used in electronic devices. Meanwhile, composite materials are a mixture of two or more materials. They usually consist of a reinforcing material in a matrix. Two outstanding types of modern composites materials used for engineering applications are fiberglass-reinforcing material in epoxy matrix and carbon fibres in epoxy material. Composites of this type have excellent corrosion resistance.

Material and Apparatus

Part A: Samples labeled A, B, C, D E, F and G, vernier caliper, weighing balance.
Part B: Samples labeled H, I, J, K, and L, 250ml beaker.
Solutions of :

ethanol/water solutions of various concentrations:
52% ethanol (density = 0.911),
38% ethanol (density = 0.9408), and
24% ethanol (density = 0.9549)

calcium chloride/water solutions of various concentrations:
6% $CaCl_2$ (density = 1.0505),
32% $CaCl_2$ (density = 1.3059), and
40% $CaCl_2$ (density = 1.3982)

Experimental Procedure

A. Density measurement

1. Measure the dimensions of each sample (length, width and thickness).
2. Weigh the sample on a weighing balance.
3. Obtain the density of the material by using the formula:

   $\rho = \frac{m}{V}$, where m = mass of object, V = volume of object.

B. Identification of Plastic Materials

1. Examine each sample and write a visual description in the proper location in your data table.
   a) Is the sample clear? Is the sample opaque? Does it have color?
   b) Flex each sample through an angle of 10^{o} to 30^{o}. Note in the data table how easy it was to flex the sample. Is it flexible or rigid? You might want to compare the various samples.
2. Pour 50 ml of 40% $CaCl_2$ (Solution 1) into a 150 ml beaker. Place each of the plastic samples in the solution. Note which of the samples sink (S) or float (F) in your data table.
3. Return the solution to the appropriate container and dry out the beaker.
4. Dry off your samples.
5. Repeat Step #2-5 with:

Solution 2:	32% calcium chloride,
Solution 3:	6% calcium chloride,
Solution 4:	24% ethanol,
Solution 5:	38% ethanol and
Solution 6:	52% ethanol.

Analysis and Questions

1. Classify the samples according to their groups (metals, polymers, ceramics, etc.).
2. Compare the measured density with obtained in the experiment with known material densities from handbooks and other technical references.
3. Find the melting temperature of each sample by referring to handbooks and other technical references.
4. State whether the materials are good electrical and heat conductors or not.
5. State whether the materials are ductile or brittle.
6. Suggest an application for each type of material identified. Give the properties or characteristics that make the material suitable for the application.

Data Sheet
Part A:

Sample	***Dimensions (mm)***					**Class**	**Material**	**Weight**	**Volume**	**Measured Density**	**Theoretical Density**
	Length	*Width*	*Inner Dia*	*Outer Dia*	*Thickness*						
A											
B											
C											
D											
E											

Part B:

Sample	**Clear / Opaque / Colour**	**Flexible / Rigid**	**Solutions for Materials (Sink (S) / Float (F))**						**Material**
			1	**2**	**3**	**4**	**5**	**6**	
F									
G									
H									
I									
J									
K									
L									
M									

Analysis:

Specimen	**Material**	**Theoretical Density**	**Melting point, T_m**	**Electrical conductivity**	**Heat conductivity**	**Brittle / Ductile**	**Application**	**Properties / Characteristics**
A								
B								
C								
D								
E								

EXPERIMENT 2: ATOMIC ARRANGEMENT IN SOLIDS

Objective

This experiment enables the student to build different models of the cubic structure, which are hard to visualize or draw. The objective is to identify atomic and interstitial positions, determine coordination number and maximum radius of interstitial atoms.

Introduction

If atoms or ions of a solid are arranged in a pattern that repeats itself in three dimensions, they form a solid which is said to have a crystal structure. The material is referred to as a crystalline solid. Atomic arrangements in crystalline solids can be described by an infinite array of points in a three-dimensional network, called space lattice. Each space lattice can be described by specifying the atom positions in a repeating unit cell. There are four basic types of unit cells: (1) simple, (2) body-centered, (3) face-centered, and (4) base-centered. Together with the 7 crystal systems, these form the 14 Bravais lattices (refer to Figure 2.1).

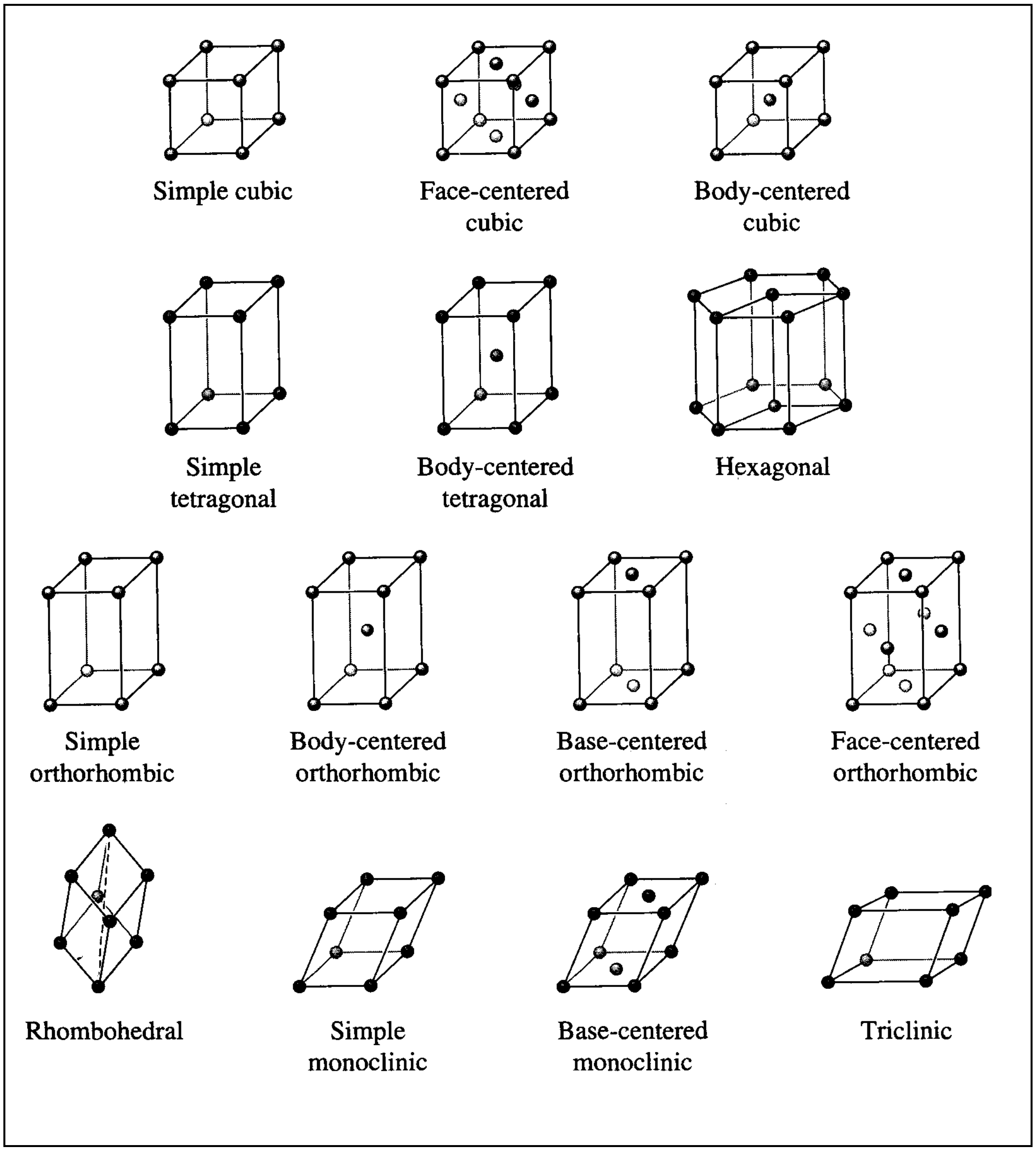


Figure 2.1. The fourteen Bravais Lattices grouped into seven crystal systems.

Material and Apparatus

Ping-pong balls, metal rods, glue.

Experimental Procedure

Simple Cubic

1. Build the model of a simple cubic structure.
2. Determine all the coordinate points of the atoms in the structure.
3. Determine the coordination number of the atom in simple cubic structure.

Face-centered Cubic

1. Build the model of a face-centered cubic structure.
2. Determine the coordination number of a corner atom.
3. There are two types of interstitial points: octahedral and tetrahedral. Determine these types of interstitial points in the structure.

Body-centered Cubic

1. Build the model of a body-centered cubic structure.
2. Determine the coordination number of a corner atom.
3. Determine the octahedral and tetrahedral interstitial points within the structure.

Hexagonal Close-packed

1. Build the model of a hexagonal close-packed structure.
2. Determine the coordination number.
3. Compare the stacking arrangement of the HCP and FCC structures.

Exercise

1. Place 3 square arrays directly over one another. Analyze the structure and determine the following:
    (a) The percentage of voids in the structure
    (b) The number of equivalent atoms in the structure
    (c) Show that the radius of the atom that can enter the interstice is 0.732 if the radius of the primary atom is unity.

2. Draw the following directions in a cubic unit cell using Miller indices: [100], [110], [201], [101], [210], [120] and [221].

3. Name the following Miller indices planes in a cubic unit cell (refer to Figures 2.2a – 2.2f):

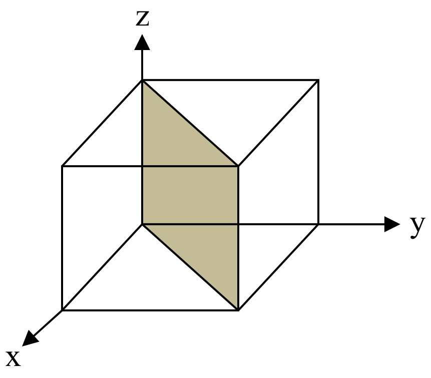


Figure 2.2a

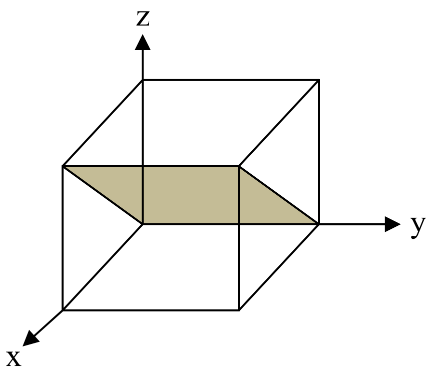


Figure 2.2b

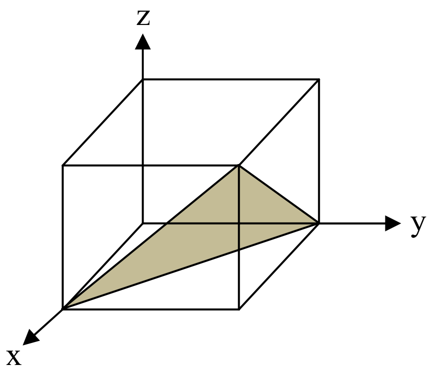


Figure 2.2c

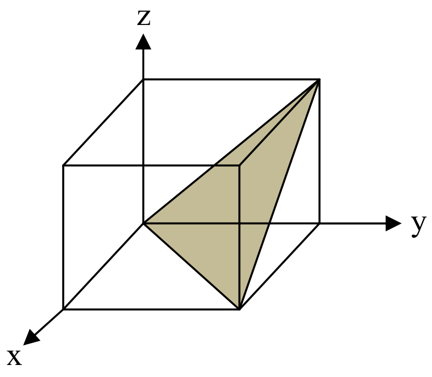


Figure 2.2d

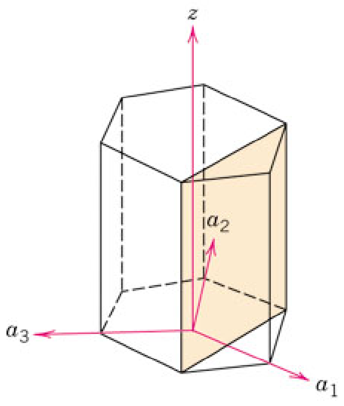


Figure 2.2e

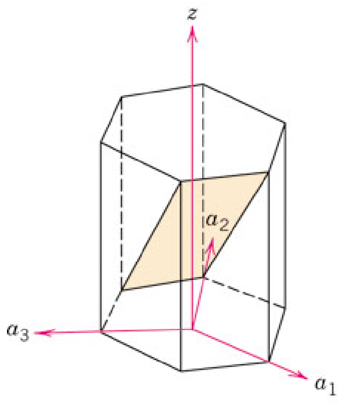


Figure 2.2f

EXPERIMENT 3: TENSILE TEST

Objectives

The objectives of the tensile test are to evaluate the strength of metals, to study its behavior under a tensile load and to obtain important parameters such as Young's Modulus, yield stress and ultimate tensile stress.

Introduction

The tensile test is the most common mechanical test to evaluate the strength of metals. It is carried out by pulling a sample to failure in a relatively short time at a constant rate.

The force (load) on the sample being tested is plotted against the corresponding strain. The force data obtained from the tensile test can be converted to engineering stress data, and a plot of engineering stress versus engineering strain can be constructed.

The mechanical properties of metals and alloys which are of engineering importance for structural design and which can be obtained from the engineering tensile test are:

1. Modulus of elasticity
2. Yield strength at 0.2% offset
3. Ultimate tensile strength
4. Percent elongation at fracture
5. Percent reduction in area at fracture

Material and Apparatus

Universal testing machine, plain carbon steel specimens.

The types of samples used for the tensile test vary considerably. For metals with a thick cross-section such as a plate, a 0.050in diameter round specimen is commonly used (see Figure 3.1(a)). For metals with thinner cross sections such as sheet, a flat specimen is used (see Figure 3.1(b)). A 2-in gage length within the specimen is the most commonly used gage length for tensile tests.

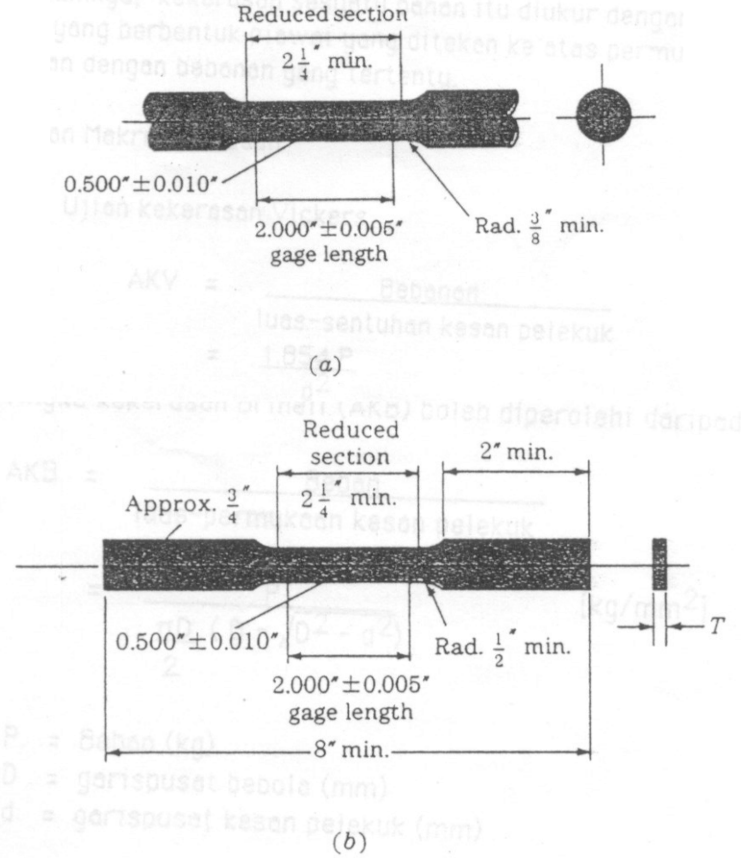


Figure 3.1 (a) Standard round tension test specimen with 2-in gage length. (b) Standard rectangular tension test specimen with 2-in gage length.

Procedure

1. Measure the original gage length and area of the specimen using a vernier caliper.
2. Carry out the tensile test for the specimen using the Universal testing machine under the supervision of the lab technician/instructor.
3. After the test is completed, obtain the percent elongation and percent reduction of area.
4. From the plot of force versus strain, obtain the graph of engineering stress versus engineering strain.
5. Obtain the Young's Modulus, ultimate tensile stress, 0.2% offset yield stress and fracture stress for the specimen.

Calculations

1. Engineering stress: $\sigma = \dfrac{F}{A_o}$
2. Engineering strain: $\varepsilon = \dfrac{l - l_o}{l_o} = \dfrac{\Delta l}{l_o} = \dfrac{\text{change in length of specimen}}{\text{original length of specimen}}$
3. Ultimate tensile stress: $\sigma_{ult} = \dfrac{F_{\max}}{A_o}$
4. Percent elongation: % elongation = $\dfrac{l - l_o}{l_o} x100\%$
5. Percent reduction in area: % reduction in area = $\dfrac{A_o - A_f}{A_o} x100\%$
6. Young's Modulus or modulus of elasticity can be obtained from the linear relationship between stress and strain in the elastic region of the engineering stress-strain diagram.

7. Yield strength is the strength at which a metal or alloy shows significant plastic deformation. Because there is no definite point on the stress-strain curve where elastic strain ends and plastic strain begins, the yield strength is chosen to be the strength when a definite amount of plastic strain has occurred, for example, 0.2% offset yield strength (see Figure 3.2).

where F = average uniaxial tensile force

A_o = original cross-sectional area

l = new length of specimen after being extended by uniaxial tensile force

l_o = original length of specimen

F_{max} = maximum tensile force

A_f = final cross-sectional area

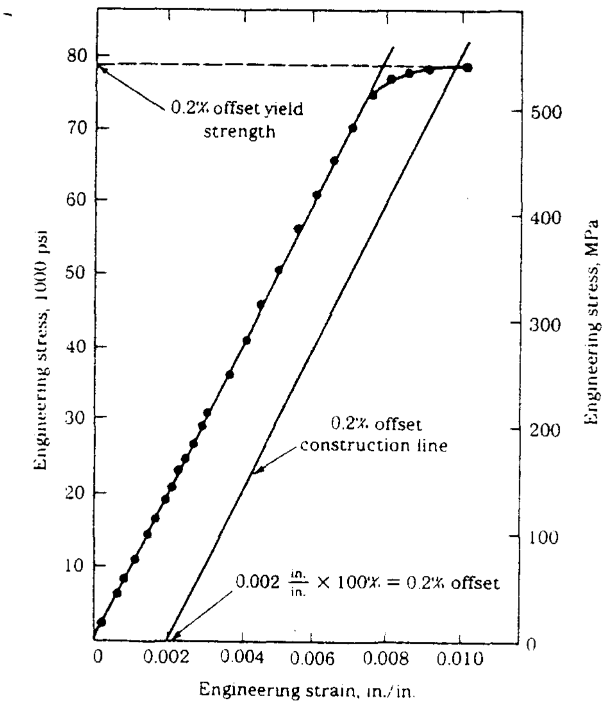


Figure 3.2. Determination of the 0.2% offset yield stress.

Questions

In your results and discussion, you should include the following:

1. Describe what you observe about the specimen after failure.
2. Compare the stress-strain graph that you have obtained for the specimen with that of an aluminium specimen. (Refer to handbooks and other technical references).
3. What are some of the factors that may affect the precision of your results in carrying out this test?

EXPERIMENT 4: HARDNESS TEST

Objectives

The objective of this experiment is to determine the hardness of a material by using a hardness tester.

Introduction

Hardness is a measure of the resistance of a metal to permanent (plastic) deformation. The hardness of a metal is measured by forcing an indenter into its surface. The indenter, which is usually a ball, pyramid, or cone, is usually made of hardened steel, tungsten carbide, or diamond, a much harder material than the material being tested. For most standard hardness tests, a known load is applied slowly by pressing the indenter at 90^o into the metal surface being tested. The indenter is withdrawn from the surface after the indentation is made. An empirical hardness number is then calculated or read off a dial, which is based on the cross-sectional area or depth of the impression. The four common hardness tests are Brinnell, Vickers, Knoop and Rockwell.

Material and Apparatus

Hardness testers: Vickers and Knoop microhardness.
Test Specimens: Steel, copper, brass, aluminium.

Experimental Procedure

The hardness tests should be carried out under the supervision of the lab technician/instructor.

General procedure:

1. Place the specimen on the vice.
2. Lower the indenter until it just touches the specimen surface.
3. Set the appropriate load for the indenter.
4. Allow the indenter to penetrate the specimen surface.
5. Load is removed and indenter is raised from specimen surface.
6. Measure the appropriate parameter (depth, length or diameter) of the indentation.
7. Compute the hardness value.
8. Repeat the above for 5 times at different locations on the specimen and compute the average value.

Calculations

1. Vickers Hardness Number (VHN)

$$\text{VHN} = \frac{1.72P}{d_1{}^2}$$

2. Knoop microhardness

$$\text{KHN} = \frac{14.2P}{l^2}$$

P = load (kg)

d_1=average diagonal length of indentation (mm)

l = length of the longest diagonal (mm)

Questions

1. Comment on the indenters used for Vickers and Knoop hardness.
2. Compare the hardness values obtained each time for the same specimen and comment on their differences.
3. Compare the hardness values obtained using Vickers and Knoop. Explain the differences observed.
4. Under what circumstances is the Knoop microhardness test usually used?

EXPERIMENT 5: FATIGUE TESTING

Objective

The objective of this experiment is to test the fatigue strength of a material subjected to repetitive or cyclic stress.

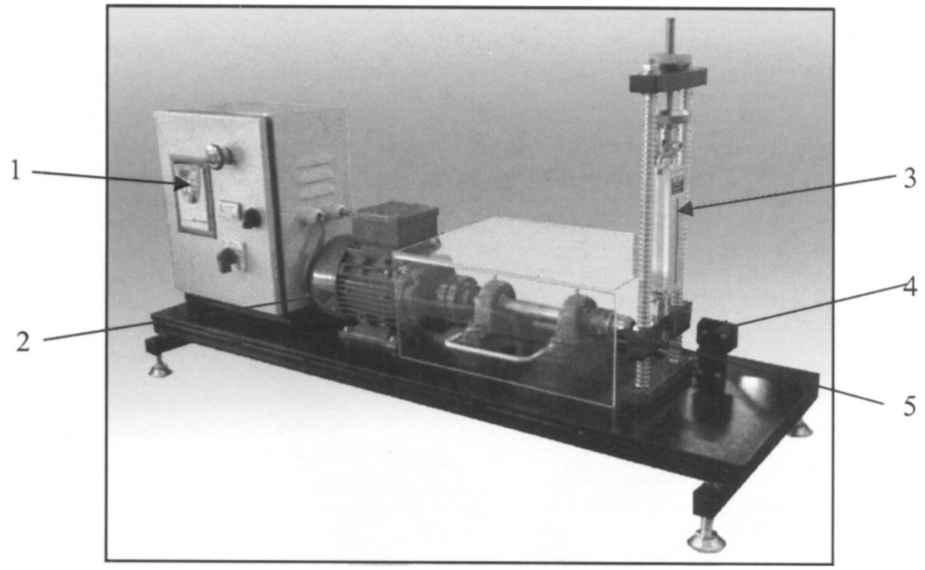
Introduction

The failure of materials under a repeated or cyclic stress is known as fatigue failure. These failures occur at a much lower stress than that which the material can withstand under the application of a single static stress. Examples of parts which fail by fatigue are commonly moving parts such as shafts, connecting rods and gears. About 80% of failures in machines are attributed to the direct action of fatigue failures. Fatigue failures usually originate at a point of stress concentration such as a sharp corner or a notch.

The most commonly used small-scale fatigue test is the rotating beam test in which a specimen is subjected to alternating compression and tension stresses of equal magnitude while being rotated. Tests are carried out on a standard specimen to find the number of stress reversals (N) required to cause a fatigue failure at a given alternating stress level (σ).

Material and Apparatus

Aluminium, mild steel and copper specimens, LS-22046 Fatigue Testing Machine (see Figure 5.1).



1. Frequency inverter 2. Three-phase motor
5. Loading scale 4. Limit switch
3. Specimen chuck holder

Figure 5.1. LS-22046 Fatigue Testing Machine

Experimental Procedure

At least three specimens loaded at different forces should be carried out to failure to determine the S-N curve. Follow the procedure outlined below:

1. Set up the Fatigue Testing Machine by plugging into a 240VAC single phase 50Hz supply.
2. Position the specimen into the shaft end first and then into the bearing end that applies force.

3. Slowly tighten the grips in the chuck with the tee key provided.
4. Set the cycle counter to zero.
5. Set the required force by turning the load nut. (Aluminium – 160N, mild steel – 90N and copper – 160N).
6. Turn on the main switch and set the required frequency of the motor.
7. Let the motor run until the specimen fails.
8. Record the number of turns before failure.
9. Repeat the experiment using a different force.
10. Plot the S-N curve on semi-log graph paper.

Calculation

Bending stress due to bending loads in a beam is defined as

$$\sigma_{bending} = \frac{My}{I}$$

Therefore, $\sigma_{Alt} = K_t \frac{My}{I}$

Where M = Moment = Force x distance = F x 0.0364 Nm

$y = 0.0045$m

$I = 3.22 \times 10^{-10}$ m^4

$K_t = 2$ (constant)

Results

Force (N)	Stress σ (N/mm^2)	Number of cycles to failure		
		Aluminium	Mild Steel	Copper
190				
160				
120				
90				
70				

Questions

1. Comment on the S-N curve obtained for different specimens. Explain why the curves appear different.
2. Compare the number of cycles to cause failure for the different types of material.
3. Examine the fracture surface of the specimens. Describe the characteristics of their fractured surfaces.
4. Briefly describe the factors that affect the fatigue strength of materials.

EXPERIMENT 6: CALIBRATION OF THERMOCOUPLE

Objectives

The objective of the experiment is to calibrate a thermocouple and the find the corresponding curve-fit correlation.

Introduction

When two dissimilar metal wires are connected with each other in a loop to form two junctions, maintained at two different temperatures, a voltage potential or electromotive force (E=emf) will be generated and the current will flow through the loop circuit. The current will be proportional to the difference in temperature between the junctions and the metals used. The higher the temperature difference, the higher is the electromotive force (emf) and the current flow in the loop. The magnitude of the emf is in the order of few milivolts. We need a precise and sensitive multimeter which can read up to microvolts to do this experiment.

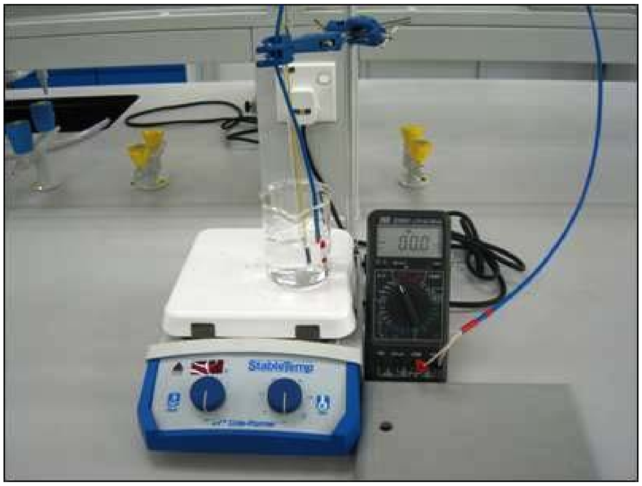


Figure 6.1 Experimental set-up for calibration of thermocouple.

Materials and Apparatus

Thermocouple, beaker, thermometer, multimeter.

Experimental Procedure

1. Assemble the apparatus as shown in Figure 6.1.
2. Turn on the hot plate to increase the temperature of the water in the beaker.
3. Heat the beaker to the desired temperature (30^{o}C to start). Switch off the hot plate once the temperature is reached.
4. Connect the ends of the thermocouple to the digital multimeter and set the multimeter to read in milivolts DC.
5. Dip one junction of the thermocouple in the thermo-bath liquid and wait for a few minutes for it to reach the steady sate (i.e. the reading on the multimeter steadies down except the last digit). Be careful to hold (tape) the sensor wire away from the circulator's propeller.
6. Note down the digital multimeter reading in milivolts (E_{MM}) and repeat steps 3 through 6 in steps of 5 degrees from 30 to 60 degrees Celsius.
7. The multimeter reading corresponds to the difference in temperature between the surroundings (room) and the bath. To calibrate the thermocouple, we have to take the room temperature into consideration to get the absolute value of temperature measured. Find the equivalent milivolt value for the room temperature from the corresponding thermocouple table (E_{RM}). Then add that milivolt value, corresponding to the room temperature, to every multimeter reading (E_{MM}). Tabulate the values.

Analysis

1. Plot the measured bath temperature values (T_B) on the y-axis against the corresponding thermocouple emf (milivolt) values (E) on the x-axis.
2. Find the slope, intercept and correlation coefficient of the curve-fitted line by any method. If the correlation coefficient is not very close to one, curve fit with higher order polynomial. Calculate the 95% uncertainty in temperature measurement of the calibrated thermocouple sensor if the obtained curve-fit correlation is used.

EXPERIMENT 7: PHASE DIAGRAM

Objectives

The objectives of this experiment are:

(a) To provide a simple practical solidification process through solidifying Pb-Sn alloys of four different compositions.
(b) To obtain cooling curves during solidification.
(c) To predict the microstructure development during slow cooling.
(d) To understand how a phase diagram is constructed.

Introduction

For a pure element, there exists a certain temperature at which the material changes from a solid phase to a liquid phase (see Figure 7.1). This temperature is called the solidification, freezing or melting temperature. Alloys do not have a single melting temperature. Instead, there is a range of temperatures over which the material is a mixture of both solid and liquid (see Figure 7.2). The extent of this temperature range depends upon the alloy's composition. The temperatures at which the alloy is 100% liquid and 100% solid are used to define this range. These temperatures are plotted against the alloy's composition and are called the liquidus and solidus lines, which form part of the phase diagram.

In this experiment, you will construct the solidus and liquidus curves for a binary alloy Pb-Sn. These curves will be found using a thermocouple to monitor the temperature of specimens of various compositions during continuous cooling. By repeating this experiment for a number of compositions, you will be able to outline the general shape of the phase diagram.

Materials and Apparatus

Crucible, tongs, bunsen burner, type-K thermocouple, strip chart recorder, cooling rack, weighing balance, gloves.

Reagent grade granulated lead and tin of the following compositions (in wt%):

1. Sample 1: 100%Pb – 0% Sn
2. Sample 2: 70% Pb – 30% Sn
3. Sample 3: 38% Pb – 62% Sn
4. Sample 4: 30% Pb – 70% Sn

Caution: Lead is toxic. Use gloves when handling the material. Do not inhale the fumes during heating.

Experimental Procedure

Heating of samples:

1. Place the Bunsen burner under the crucible using a pair of tongs and heat until the lead melts. Be careful not to overheat.
2. After the metal has melted, turn off the burner and allow the crucible to slowly cool on a cooling rack.
3. Insert the thermocouple into the melt and chart the voltage output of the thermocouple at an interval of every 5 seconds. The thermocouple must be located in the middle of the molten metal.
4. After the sample has cooled to the point where no further phase transformations are expected, remelt the sample to remove the thermocouple.
5. Repeat the procedure for the remaining samples.

Analysis

1. Analyze the cooling curves of all samples to determine the transformation temperatures for each composition and construct a table to clearly represent these data.
2. Next, construct a phase diagram from the data. Clearly and neatly label all important features, e.g. liquidus, solidus, eutectic point and phase regions.
3. Include sketches of the microstructure that you would expect for each sample composition during cooling.

Questions

1. Compare your results with the phase diagram in Figure 7.3. If there are any differences, explain them.
2. The cooling curves for some of the mixtures may show a dip in temperature as shown in Figure 7.1. Explain why this occurs.
3. Include sketches of the microstructures (expected phases) for each of the sample compositions during cooling.
4. Based on Figure 7.3, determine the phases and amount of phases present in the 60%Pb – 40%Sn and 40%Pb – 60%Sn and eutectic compositions using the Lever Rule.
5. Comment on the significance of the solvus lines.

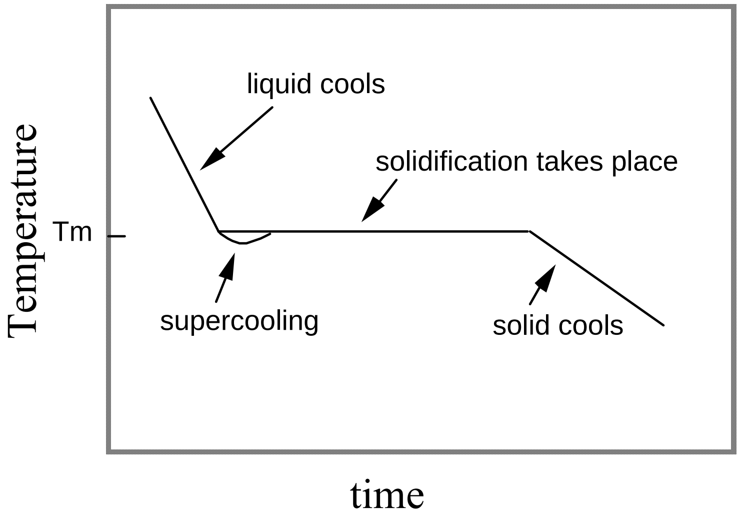


Figure 7.1. Near equilibrium cooling cure for a pure metal or a eutectic alloy. Solidification takes place at a single temperature.

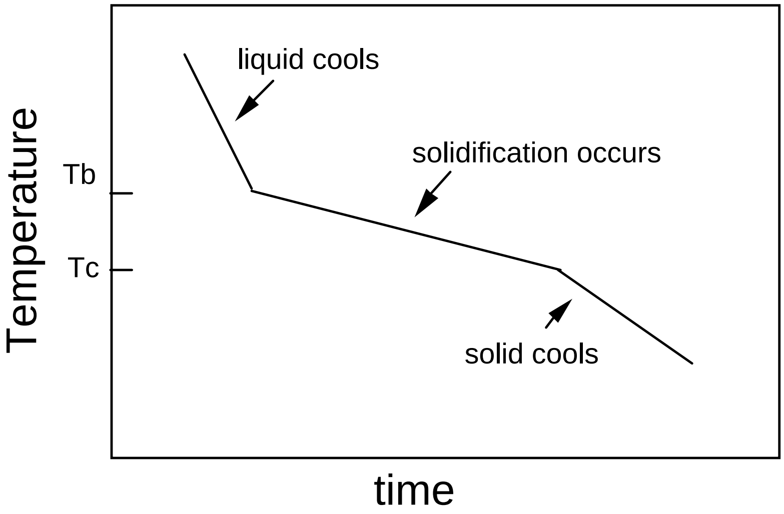


Figure 7.2. The cooling curve characteristic of a binary alloy where solidification takes place over a range of temperatures. T_b is the temperature at which solidification begins and T_c is the temperature at which solidification is complete.

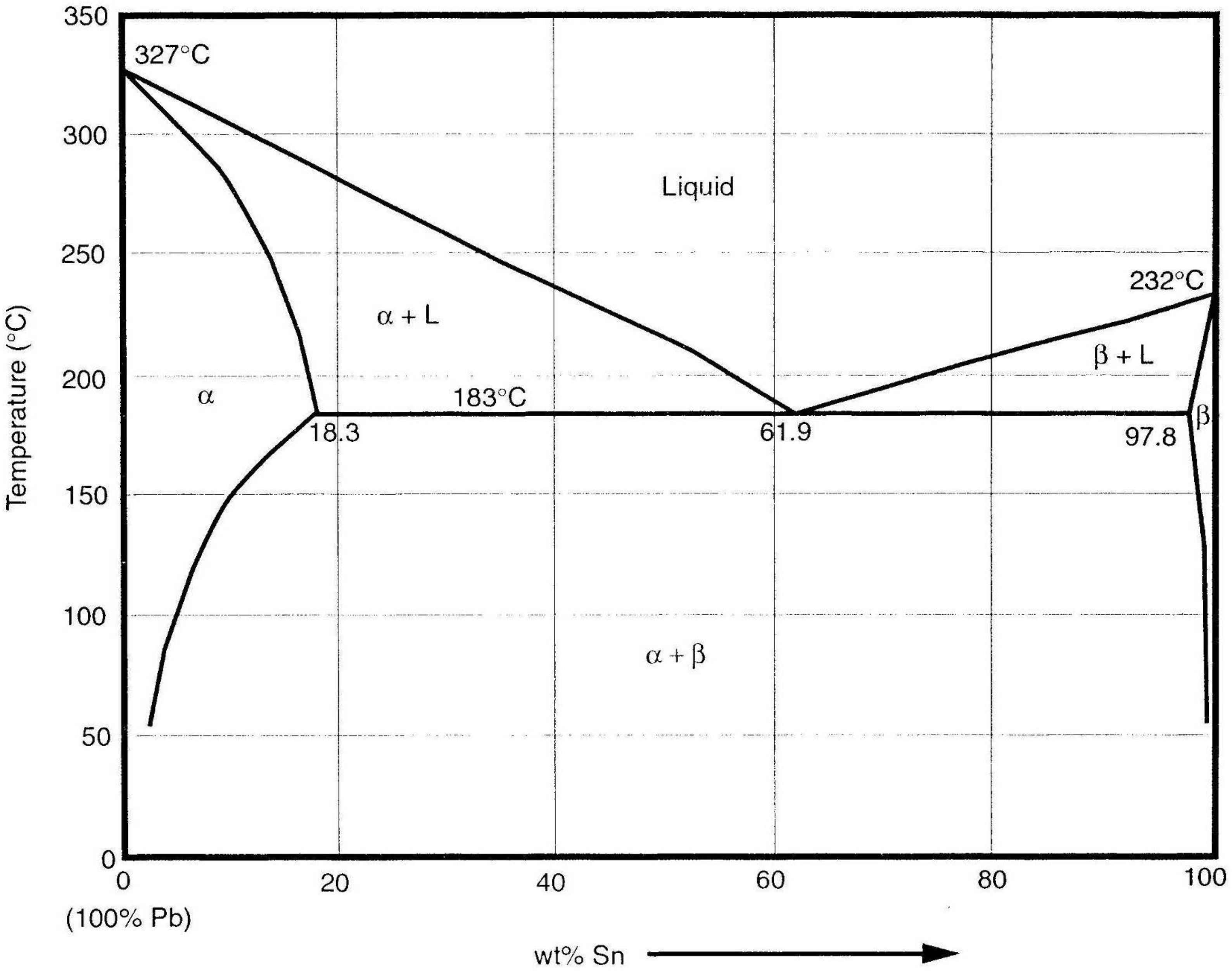


Figure 7.3. The Pb-Sn equilibrium phase diagram.

EXPERIMENT 8: METALLOGRAPHIC TECHNIQUES AND MICROSCOPY

Objectives

In this experiment, the student will learn to prepare metallographic samples for microscopic examination. Techniques of grinding, polishing and etching will be gained hands-on. Finally the student has to identify the microstructure of the sample through the use of a microscope.

Introduction

Preparation of metallographic specimens generally requires five major operations: (a) sectioning, (b) mounting, (c) grinding, (d) polishing and (e) etching.

(a) Sectioning

Abrasive-wheel cutting is by far the most widely used sectioning method in metallographic laboratories. However, abrasive-wheel cutting may produce deformation damage. This can be minimized using thin cut-off wheels.

(b) Mounting

The primary purpose of mounting is for convenience in handling specimens of difficult shapes. Common mounting methods include compression mounting (hot mounting) and cold mounting.

(c) Grinding

Grinding is accomplished by abrading the specimen surface through a sequence of operations using progressively finer abrasive grit. Grinding should proceed with coarse grit to establish an initial flat surface and to remove the effects of sectioning.

(d) Polishing

Polishing is the final step in producing a surface that is flat, scratch-free and mirror-like in appearance. This is necessary for subsequent metallographic examination. Various methods may be employed such as mechanical, electrolytic and chemical polishing.

(e) Etching

Metallographic etching is used to reveal particular structural characteristics of a material that are not evident in the as-polished condition. The principle of etching is based on the preferential attack (different rates of solution of the phases in the etchant) or preferential staining of one or more phases because of differences in chemical composition and orientation.

Materials and Apparatus

Cold mounting kit complete for sample preparation, grinder/polisher machine, etchant, cotton buds, dryer, beaker, microscope, metal specimens labeled A, B, C, D, E, F, G, H, I and J.

Experimental Procedure

Mounting:

1. Measure the appropriate ratio of resin and hardener according to manufacturer's recommendation.
2. Mix the two together in a sample cup using a mixing stick. Try to avoid introducing air bubbles during mixing.
3. Place the sample in the mould and pour the mixture in. Ensure the level of the mixture sufficiently covers the level of the specimen.
4. Allow sample to set within the mould according to manufacturer's recommended time.

Manual Grinding:

1. Turn on the tap of the grinder. Place the sample face down onto grinding paper and abrade in one direction only. Start with abrasive paper of grit size 240.
2. Continue the action until a flat surface is achieved.
3. Proceed to the next station with abrasive paper of grit size 400. Repeat the abrading operation with the sample, still face down, but in the orthogonal direction. Continue until scratches from the previous operation is totally removed.
4. Repeat step 3 progressively with abrasive paper of grit size 600 and 1000.

Manual Polishing:

1. Turn on the polisher and adjust the speed to low.
2. Drop some amount of polycrytsalline suspension (6µm) on the Texpan polishing nap.
3. Place the sample face down onto the polishing plate and apply even pressure by hand. Polish the sample until it has a mirror-like appearance and scratch free.
4. Wash and dry the sample.
5. Repeat steps above using finer suspension (1µm).
6. After that, repeat steps above using alumina suspension (0.3µm and 0.05µm) on microcloth.

Chemical Etching:

1. Pour a small amount of etchant into a beaker.
2. Wet the tip of a cotton bud by dipping into the etchant.

3. Smear the surface of the sample using the cotton bud for a short period of time.
4. Wash sample over running water and alcohol, then dry.
5. Examine the microstructure of the sample using an optical microscope. If microstructure is not revealed, repeat steps 2 to 5.

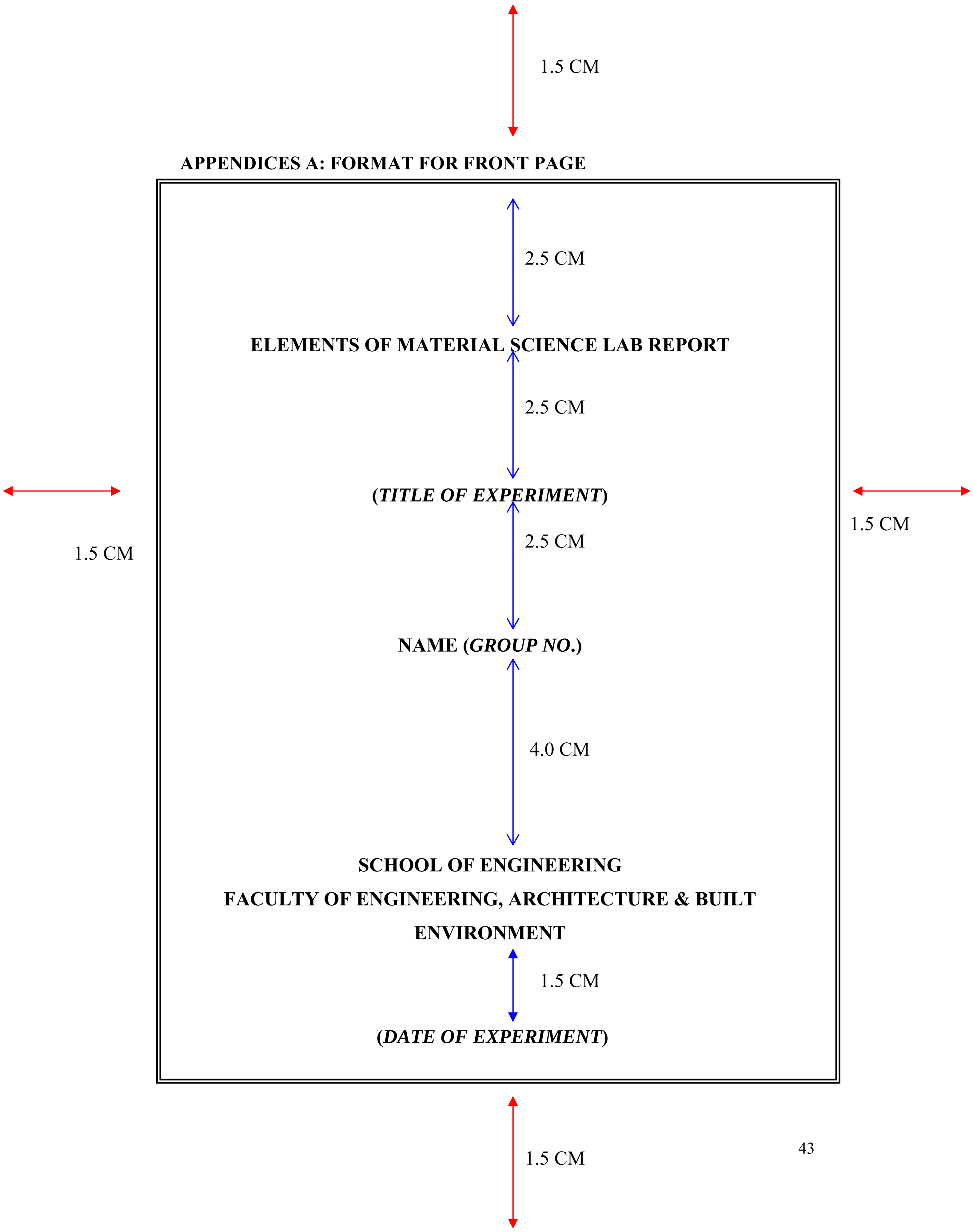
Microscopy:

1. Turn on the microscope.
2. Turn the objective lens to the lowest magnification.
3. Place the sample on the stage by adjusting the stage travel distance.
4. Adjust the eyepiece to suit your eyes.
5. Adjust the focus of the object by turning the focusing knob.
6. Note your observations through the microscope.

Analysis

1. Sketch the microstructure of the specimen, taking note of any defects present, the morphology and other characteristic features (e.g. grain size, orientation, segregation, etc.). Comment on these features.
2. Using the microscope, examine the prepared specimens labeled B, C, D, E, F, G, H, I and J. Sketch and determine the microstructure of each specimen, and indicate a suitable etchant that can be used to reveal its characteristics.

APPENDICES A: FORMAT FOR FRONT PAGE



ELEMENTS OF MATERIAL SCIENCE LAB REPORT

(*TITLE OF EXPERIMENT*)

NAME (*GROUP NO.*)

SCHOOL OF ENGINEERING
FACULTY OF ENGINEERING, ARCHITECTURE & BUILT ENVIRONMENT

(*DATE OF EXPERIMENT*)

EXAMPLE OF COVER PAGE

ELEMENTS OF MATERIAL SCIENCE LAB REPORT

EXPERIMENT 1: IDENTIFICATION OF ENGINEERING MATERIALS

JOHN SMITH (GROUP NO. 1)

SCHOOL OF ENGINEERING
FACULTY OF ENGINEERING, ARCHITECTURE & BUILT ENVIRONMENT

10 JANUARY 2011

APPENDIX B: FORMAT FOR TABLE OF CONTENT

2.5 CM

TABLE OF CONTENT

4 SPACE

1.0	**Introduction**	Page no.
2.0	**Material & Methodology**	
3.0	**Results and Discussion**	
4.0	**Conclusion**	
5.0	**Limitation of Experiment**	
	Reference	

APPENDICES C: CITATION OF REFERENCES

Reference Style

The details of the references cited in the text, published or unpublished should be located in the List of References. The list should be placed at the end of the report, compiled alphabetically (Harvard System).

Citing in the Text

The references cited in the text should be indicated using the name of the author and the date of publication. Examples are as follow:

(a) If the name of an author is written as part of a sentence, the year published should be written in parentheses.
"Works by Yao (1993) have shown that in order to maintain the behavioural link between the offsprings and their parents, the use of crossover operator should be avoided."

(b) If the name of an author is not written as part of a sentence, both the name and year published should be written in parentheses.
"ANN offers useful properties and capabilities such as non-linearity, input and output mapping, adaptability and fault tolerance among others (Haykin, 1999)."

(c) If there are two authors for a cited reference, both names should be written.
"In designing the model for non-linear system, the parsimonious principle (Soderstrom and Stoica, 1989) is critical because a nonlinear model involves an excessive number of parameters." "Syu and Chang (1999) successfully used neural networks to adaptively control *Penicillin acylase* fermentation."

(d) If there are more than three authors for a cited reference, use *et al.* after the name of the first author.
"The algorithm can be calculated by applying Gram-Schmidt procedures as described by Korenberg *et al*. (1988)."

(e) If more than one reference materials by the same author in a same year are cited, use small letter alphabets (a, b, c, and so on) to distinguish them.
"Some of the basic principles widely used by many researchers are Lagrange-Euler (LE) equations (Uicker, 1965; Bejczy and Paul, 1981), Newton-Euler (NE) equations (Luh *et al*., 1980a) and d'Alembert (GD) equations (Lee *et al*., 1983)."
"Luh *et al.* (1980b) presented an example of an acceleration control of robot arm/manipulator."

Writing Style for Authors' Names in the List of References

Generally, authors' names are listed using surname followed by their initials. The followings are examples of writing style according to the name of the author:

(i) Single and multiple authors

Example (single author):

Veres, S. M. (1990). *Structure Selection of Stochastic Dynamic Systems*. New York: Gordon and Breach Science Publishers.

Example (two or more authors):

Soderstrom, T., and Stoica, P. (1989). *System Identification*. United Kingdom: Prentice Hall International Ltd.

Luh, J. Y. S., Walker, M. W., and Paul, R. P. (1980b). Resolved-Acceleration Control of Mechanical Manipulators. *IEEE Trans. Automatic Control*. 25(3): 468-474.

(ii) Editor

Example:

Martin, A. M. (Ed.) (1991). *Peat as an Agent in Biological Degradation of Waste*. London: Elsevier.

Lees, R. H. and Thomas T. R. (Eds.) (1974). *Chemical Nomenclature Usage*. Chichester: Ellis Horwood.

(iii) Corporate author/editor

Example:

Engineers Joint Council (1969). *Thesaurus of Engineering and Scientific Terms*. New York: Engineers Joint Council.

Writing Style for Various Types of Publication Materials in the List of References

Frequently, different types of publication materials are cited in a report. The style of writing details on cited publication should be as follows:

(i) Book

Author (Year). *Title*. (Edition). Placed published: Publisher.

Example:

Theusen, G. J. and Fabrycky, W. J. (1984). *Engineering Economy*. (6th ed.) Englewood Cliffs, N. J.: Prentice Hall.

(ii) Article in a book

Author of the article (Year). *Title of the article*. In author or editor of the book. *Title of the book*. (page). Place published: Publisher.

Example:

Hussein, S. B., Jamaluddin, H., Mailah, M. and Zalzala, A. M. S. (2000). An Evolutionary Neural Network Controller for Intelligent Active Force Control. In Parmee, I. C. (Ed.) *Evolutionary Design and Manufacturing* (pp. 351 –362). London: Springer-Verlag.

(iii) Journal articles

A. Print format

Author (Year). Title of the article. *Title of the Journal*. Volume (Issue no.), page.

Example:

Billings. S. A. (1980). Identification of Nonlinear Systems: A survey. *Proc. IEE, Part D.* 127(6), 272-284.

B. Electronic format

Author (Year). Title of the article. *Title of the Journal*. Volume (Issue no.), page. Publisher.

Example:

Borman, W. C. (1993). Role of Early Supervisory Experience in Supervisor Performance. *Journal of Applied Psychology*, 78, 443 – 449. American Psychology Association.

(iv) Conference articles

Author (Year). Title of the article. *Name of the conference*. Date of the conference. Place, page.

Example:

Sheta, A. F. and De Jong, K. (1996). Parameter Estimation of Nonlinear Systems in Noisy Environments Using Genetic Algorithms. *Proceedings of the 1996 IEEE International Symposium on Intelligent Control*. 15-18 September. Dearborn, Michigan: IEEE, 360 - 365.

(v) Thesis

Author (Year). *Title of the thesis*. Thesis award. Place published.

Example:

Adnan bin Hassan (2002). *On-line Recognition of Developing Control Chart Patterns*. Ph.D. Thesis. Universiti Teknologi Malaysia, Skudai.

(vi) Legislations

Name of the country (year). *Title of the legislation*. Legislation number.

Example:

Malaysia (1983). *Perintah Monumen Lama dan Tapak Tanah Bersejarah*. P.U.(A)41 1983.

(vii) Standards

Name of the institution (Year). *Standard number*. Place published: Publisher.

Example:

British Standards Institution (1990). *B.S. 764*. London: British Standards Institution.

(viii) Patent

Print format

Author (Year). *Patent number*. Place published: Official source.

Example:

Smith, I. M. (1988). *U.S. Patent No. 123, 445*. Washington DC: U.S. Patent and Trademark Office.

Electronic format

Author (Year). *Patent number*. Retrieved on date, year, from URL address of the patent database.

Example:

Ulrich, K. (2001). *European Patent No. EP1162184.* Retrieved on March 7, 2002, from fttp://ep.espacenet.com/

(ix) Brochure

Name of organization (Year). *Title* [Brochure]. Place published: Publisher.

Example:

Research and Training Center (1993). *Guidelines for Reporting and Writing About People with Disabilities.* [Brochure]. Lawrance, KS: Macmillan.

(x) Measured drawings

Author (Year). *Title*. [Measured drawing]. Name of organization: Place published.

Example:

Salim Man (1989). *Pengisi Sekam ke Dalam Kontena Penyimpan: Pandangan Isometrik.* [Lukisan Teknik]. Universiti Teknologi Malaysia: Skudai.

(xi) Unpublished materials

Author (Year). *Title*. Unpublished note, Name of organization.

(xii) Newspaper article

A. Print format, no author

Title of article. (Year, date). *Name of newspaper*, page.

Example:

Gearing up to meet new challenges. (2000, February 22). *The Star*. p. 2.

B. Print format, with author

Author. (Year, date). Title of article. *Name of newspaper*, page.

Example:

Izatun Shari (2000, April 18). K-economy: draft out in October. *New Straits Times*. p. 2-4.

C. Electronic format

Author. (Year, date). Title of article. *Name of newspaper*, Retrieved date, year, from URL address of the newspaper.

Example:

Rosmawati Mion (2006, June 17). Sindiket judi haram tumpas. *Utusan Malaysia.* Retrieved June 19, 2006, from http://www.utusan.com.my

(xii) Magazine

Author. (Year). Title of article. *Name of magazine*, Volume/Issue no., page

Example:

Smith, B. L. (1994). Biofeedback. *Science,* 62, 673 – 675.

(xiii) CD-ROM

Author. (Year). Title of article. [CD-ROM]. *Title of Journal,*, Volume, page. Publisher.

Example:

Ivry, R. B. (1995). Perception and production of temporal intervals across a range of durations. [CD-ROM]. *Journal of Experimental Psychology*, 21, 3-18. American Psychological Association.